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TECHNICAL PROBLEM

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[Problem(s) to be Solved by the Invention] It may be able to be imagined that it can carry out a melt blow easily on the conditions near a polyethylene terephthalate fundamentally since the molecular structure of polytrimethylene terphthalate is the structure which introduced one methylene into the ethylene portion of a polyethylene terephthalate. However, polytrimethylene terphthalate has difference, like thermal stability with a very quick crystal speed contains many oligomer and low-molecular-weight impurities in a low and a polymerization object considerably as compared with a polyethylene terephthalate, and only a crude web is obtained even if it applies the manufacture conditions in the case of being a polyethylene terephthalate as it is.

[0006] The technical problem of this invention is applying the outstanding performance which polytrimethylene terphthalate's has to a super-thin fiber web, pulling out a new performance, and establishing the industrial manufacturing method. Consequently, the polytrimethylene terphthalate super-thin fiber web was excellent in flexibility and wear nature, could be dyed the dark color by the ordinary pressure, and found out the bird clapper to the web with few polymer balls. Although the polyester super-thin fiber web by the melt blowing method for could dye it the dark color by the ordinary pressure, and moreover having excelled in robustness is very useful to simplification of a dyeing process etc., the present condition is that there is no such [ until now ] thing parenchyma.

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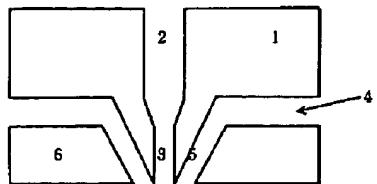
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DRAWINGS

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[Drawing 1]

FIG 1



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DESCRIPTION OF DRAWINGS

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[Brief Description of the Drawings]

[Drawing 1] It is drawing on which the die circumference of the melt blow equipment explained by this invention was drawn roughly.

[Description of Notations]

- 1 Melt Blow Die
- 2 Melting Polymer Induction
- 3 Orifice-like Nozzle
- 4 Heating Gas Induction
- 5 Heating Gas Jet Slit
- 6 Lip

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PRIOR ART

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[Description of the Prior Art] About the spinning method of the polymer by the melt blowing method Industrial - and - engineering chemistry The 48th volume, No. 8, 1956 It is indicated by 1342-1346 pages and the super-thin fiber web which used the polyethylene terephthalate in it is introduced. It or subsequent ones, a polyethylene-terephthalate homopolymer, a polyethylene-terephthalate copolymer, Many patent application about the super-thin fiber web by the melt blowing method using the polybutylene terephthalate is made (JP,53-65471,A, JP,63-53309,B, JP,3-8855,A, JP,4-2850,A).

[0003] The polytrimethylene terphthalate which is made to carry out the polycondensation of the trimethylene glycol (1, 3-propanediol) to the lower-alcohol ester of the terephthalic acid represented by a terephthalic acid or the dimethyl terephthalate on the other hand, and is obtained is the epoch-making polymer having the outstanding elastic-recovery nature, a low elastic modulus (soft feeling), a property similar to a polyamide called dyeable, and lightfastness, heat set nature, dimensional stability and a performance similar to a polyethylene terephthalate called a low water absorption. Taking advantage of the feature, it is applied to the BCF carpet, the brush, the tennis gut, etc. (JP,9-3724,A, JP,8-173244,A, JP,5-262862,A).

[0004] If the melt blow super-thin fiber web using polytrimethylene terphthalate is obtained, although a web with the new feature is expectable, the present condition is that such [ until now ] a proposal is not made. Of course, in application of the polyester web by melt blow, the feature, and the industrial usefulness or industrial manufacturing method of a certain thing are not shown for what is shown as one of the examples of polymer by which many TORITORI methylene terephthalate was enumerated as an example of polyester.

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**TECHNICAL FIELD**

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[The technical field to which invention belongs] this invention relates to the polytrimethylene terephthalate super-thin fiber web by the melt blowing method. Furthermore, in detail, it excels in flexibility and wear nature, and can be dyed a dark color by the ordinary pressure, and a polymer ball is related with the polytrimethylene terephthalate super-thin fiber web by the few melt blowing method.

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

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[0002]

[Description of the Prior Art] About the spinning method of the polymer by the melt blowing method Industrial - and - engineering chemistry The 48th volume, No. 8, 1956 It is indicated by 1342-1346 pages and the super-thin fiber web which used the polyethylene terephthalate in it is introduced. It or subsequent ones, a polyethylene-terephthalate homopolymer, a polyethylene-terephthalate copolymer, Many patent application about the super-thin fiber web by the melt blowing method using the polybutylene terephthalate is made (JP,53-65471,A, JP,63-53309,B, JP,3-8855,A, JP,4-2850,A).

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[0005]

[Problem(s) to be Solved by the Invention] It may be able to be imagined that it can carry out a melt blow easily on the conditions near a polyethylene terephthalate fundamentally since the molecular structure of polytrimethylene terephthalate is the structure which introduced one methylene into the ethylene portion of a polyethylene terephthalate. However, polytrimethylene terephthalate has difference, like thermal stability with a very quick crystal speed contains many oligomer and low-molecular-weight impurities in a low and a polymerization object considerably as compared with a polyethylene terephthalate, and only a crude web is obtained even if it applies the manufacture conditions in the case of being a polyethylene terephthalate as it is.

[0006] The technical problem of this invention is applying the outstanding performance which polytrimethylene terephthalate's has to a super-thin fiber web, pulling out a new performance, and establishing the industrial manufacturing method. Consequently, the polytrimethylene terephthalate super-thin fiber web was excellent in flexibility and wear nature, could be dyed the dark color by the ordinary pressure, and found out the bird clapper to the web with few polymer balls. Although the polyester super-thin fiber web by the melt blowing method for could dye it the dark color by the ordinary pressure, and moreover having excelled in robustness is very useful to simplification of a dyeing process etc., the present condition is that there is no such [ until now ] thing parenchyma.

[0007]

[Means for Solving the Problem] That is, this invention is a super-thin fiber web which consists of polytrimethylene terephthalate substantially, and offers the polyester super-thin fiber web which serves as a 0.8-5-micrometer diameter of average fiber from the super-thin fiber group which has the limiting viscosity of 0.4-1.0. The polymer used for this invention is polytrimethylene terephthalate which carries out the polycondensation of a terephthalic acid and the 1 and 3-propanediol substantially, and is obtained. Even if it is a polytrimethylene terephthalate homopolymer that it is substantial in this invention, it is shown that you may be the polytrimethylene terephthalate copolymer shown below. namely, the range which does not spoil the purpose and effect of this invention -- glycol components, such as acid components, such as an isophthalic acid, a succinic acid, an adipic acid, 2, 6-naphthalene dicarboxylic acid, and 5-sulfoisophthalate tetrabutyl POSUHONIUMU salt, and 1, 4-butanediol, 1, 6-hexanediol, cyclohexane dimethanol, epsilon-caprolactone, 4-hydroxybenzoic acid, polyoxyethylene glycol, a polytetramethylene glycol, etc. -- less than [ 10wt% ] -- copolymerization may be carried out

[0008] Moreover, to polytrimethylene terephthalate, the need may be accepted, and various kinds of additives, for example, a

flatting, a thermostabilizer, a defoaming agent, a ready coloring material, a flame retarder, an antioxidant, an ultraviolet ray absorbent, an infrared-absorption agent, a crystalline-nucleus agent, a fluorescent brightener, etc. may be copolymerized or mixed. As for the polymer used for this invention, it is desirable that the content of the oligomer of trimethyleneterephthalate is less than [ 3wt% ], it becomes possible to avoid an on-the-strength fall by this, and also required spinning stability is industrially securable. in addition, the oligomer to which 2-4 trimethyleneterephthalate units were usually connected with trimethyleneterephthalate oligomer -- it is -- a line -- you may be a cyclic structure even if it is structure

[0009] In exceeding 3wt(s)% , when carrying out spinning, oligomer deposits in the circumference of a nozzle, and it becomes easy to generate a polymer ball, for example. In order to perform spinning to stability for a long time, less than [ 1.5wt% ] is less than [ 1wt% ] desirable still more preferably. furthermore -- the point that the fluff of the obtained fiber decreases -- less than 0.5wt% ] -- less than [ 0.3wt% ] is desirable and still more preferably natural -- an ideal target is non-\*\* In addition, a polymer ball is the cob-like polymer generated in the edge and center section of the ball-like polymer which has a diameter with a diameter [ of web composition fiber ] of about about 10 to 500 times, or fiber. After observing this polymer ball using a microscope, or giving the means of a press, a calender, and confounding processing and others to remaining as it is or a web for a web and raising the fiber density, it is detectable by dyeing this. If a polymer ball exists mostly, the use of the super-thin fiber web obtained will be restricted greatly, and it will become impossible especially as a base fabric for artificial leather using it.

[0010] Furthermore, into the polymer used for this invention, it is desirable that the content of the with a molecular weight of 300 or less organic substance is less than [ 1wt% ], and it does not color within the limits of this, or the performance of excelling in lightfastness can be secured. The with a molecular weight of 300 or less said here organic substance is the organic substance by which copolymerization is not carried out to polymer. According to examination of this invention persons, as the with a molecular weight of 300 or less organic substance, allyl alcohol, an acrolein, 2-butanol, a hexanol, a heptanol, a glycidyl methyl ether, the oxy-propylmethyl ether, etc. existed, and it found out that the total amount of these compounds had big influence on a moldability, product durability, and weatherability. When the content of the with a molecular weight of 300 or less organic substance exceeds 1wt%, it will become or a polymer ball will become being easy to generate what it is easy to color with light. Preferably, the content of the with a molecular weight of 300 or less organic substance is 5000 ppm or less, and is 1000 ppm or less especially preferably. Of course, it is non-\*\* ideally.

[0011] As the melting point of the polymer used for this invention, it is desirable that it is 227 degrees C or more. With the melting point, it is defined in 220-250 degrees C as the peak value of the peak considered to be dissolution here. When two or more dissolution peaks exist (a shoulder peak is also included), let the peak of low temperature be the melting point. At less than 227 degrees C, a weatherproof fall becomes easy for the melting point to take place. For example, if polytrimethylene terphthalate is manufactured at once and solid state polymerization of the polymer is carried out at about 200 degrees C, the content of oligomer can be reduced greatly. However, if solid state polymerization is performed, the melting point of raw material polymer falls greatly, and will be in the state where no less than 225 degrees C are not fulfilled. Into such polymer, copolymerization of the screw-3-hydroxypropyl ether which a trimethylene glycol dimerizes and generates is carried out in large quantities, the amount of end carboxyl groups increases, consequently spinning stability and weatherability tend to fall. As the melting point of desirable polymer, it is 230 degrees C or more, and is 233 degrees C or more still more preferably.

[0012] 0.4-1.5 are desirable still more desirable, and the limiting viscosity [ $\eta$ ] of the polymer used for this invention is 0.7-1.2. In this range, fiber excellent in intensity and spinning nature can be obtained. The limiting viscosity [ $\eta$ ] in this invention is a value calculated based on the following definition formula.

$[\eta] = \lim_{C \rightarrow 0} (C/x(\eta_{\text{ar}} - 1))$

$\eta_{\text{ar}}$  in a  $C > 0$  definition formula is the value which broke the 35-degree C viscosity of the diluted solution of the sample which dissolved by o-chlorophenol of 98% or more of purity by viscosity of the above-mentioned solvent itself measured at the same temperature, and is defined as relative viscosity. Moreover,  $C$  is a solute weight value by the gram unit in the 100ml of the above-mentioned solutions.

[0013] When limiting viscosity is less than 0.4, since the molecular weight of polymer is too low, an on-the-strength manifestation becomes difficult. Conversely, since melt viscosity is too high and stable spinning is not made when limiting viscosity exceeds 1.5, it is not desirable. one sort of metal acetate [ if an example desirable as a process of the polymer used for this invention is given, will use a terephthalic acid or a dimethyl terephthalate as a raw material, and / this / trimethylene glycol ], such as a calcium acetate, a magnesium acetate, zinc acetate, a cobaltous acetate, and manganese acetate, or two sorts or more -- 0.03 - 0.1wt% -- in addition, screw hydroxypropyl terephthalate is obtained at 90 - 98% of rates of an ester interchange under an ordinary pressure or pressurization Thus, in order to make the purpose of this invention attain, it is desirable to use the acetate of metals other than transition metals. next, one sort of polycondensation catalysts, such as titanium tetrapod isopropanal PIKISHIDO, titanium tetrapod butoxide, and an antimony trioxide, or two sorts or more -- 0.03 - 0.15wt% -- desirable -- 0.03 - 0.1wt% -- it adds and is made to react under reduced pressure at 250-270 degrees C The arbitrary stages of a polymerization and putting in a stabilizer before a polycondensation reaction preferably have desirable white degree, amount of polytrimethylene terphthalate oligomer, and molecular weight of a resin constituent from a viewpoint which can control the 300 or less organic amount of resources in the amount of specification. As a stabilizer in this case, pentavalence or/, and trivalent phosphorus compounds and a hindered phenol system compound are desirable.

[0014] As pentavalence or/, and trivalent phosphorus compounds, trimethyl phosphate, triethyl phosphate, tributyl phosphate, triphenyl phosphate, trimethyl phosphite, triethyl phosphite, tributyl phosphite, triphenyl phosphite, etc. are mentioned, and trimethyl phosphite is desirable especially. A hindered phenol system compound is a phenol system derivative which has the substituent which has steric hindrance in the adjoining position of a phenol system hydroxyl group, and is a compound which has

one or more ester combination in a molecule.

[0015] Specifically Pentaerythritol-tetrakis [3 (3, 5-G tert butyl-4-hydroxyphenyl) Propionate], 1, 1, 3-tris (2-methyl-4-hydroxy-5-tert-butylphenyl) butane, 1, 3, 5-trimethyl -2, 4, 6-tris (3, 5-G tert-butyl-4-hydroxy benzyl) benzene, 3, 9-screw {2-[3-(3-tert-butyl-4-hydroxy-5-methylphenyl) propionyloxy]-1 and 1-dimethyl ethyl}-tetraoxaspiro [ 2, 4, 8, and 10-] [5, 5] undecane, 1, 3, 5-tris (4-tert-butyl-3-hydroxy-2, 6-dimethylbenzene) isophthalic acid, A triethyl glycol-screw [3 (3-tert-butyl-5-methyl-4-hydroxyphenyl) Propionate], 1, a 6-hexandiol-screw [3-(3, 5-G tert-butyl-4-hydroxyphenyl) propionate], 2 Two - Thio-diethylene-screw [3 (3 5-G tert butyl-4-hydroxyphenyl) Propionate] octadecyl-3-(3, 5-G tert-butyl-4-hydroxyphenyl) propionate] can be illustrated. Pentaerythritol-tetrakis [3 (3, 5-G tert butyl-4-hydroxyphenyl) Propionate] is desirable especially.

[0016] It has the property generally shown below in the polymer obtained by the above-mentioned method. The content of the screw-3-hydroxypropyl ether by which copolymerization was carried out is less than [ 0.1wt% ]. Moreover, when b value shows a hue, depending on 10 or less and the case, it is three or less. Moreover, the amount of end carboxyl groups is 10-35mg Eq/kg. The fiber which constitutes the polyester super-thin fiber web of this invention is the range of the 0.8-5-micrometer diameter of average fiber, and is mixed fiber which has the moderate diameter distribution of fiber. While the intensity of the fiber from which the diameter of average fiber is obtained by less than 0.8 micrometers becomes inadequate, the fall of coloring nature and robustness takes place. On the other hand, if 5 micrometers is exceeded, it will become only the web which gives a scarce crude feel to flexibility. Moreover, since this super-thin fiber has the very small diameter of fiber, although it is difficult to measure the average length of fiber, in the case of 30mm or more and many, about 70-350mm can be presumed.

[0017] Generally the amount of eyes of the polyester super-thin fiber web of this invention is 5 - 200 g/m<sup>2</sup>, although it can set up arbitrarily by the use. It is a range. The polyester super-thin fiber web of this invention has the limiting viscosity of 0.4-1.0. For this reason, its intensity is high although the web obtained is rich in flexibility. Less than in 0.4, intensity is low and a polymer ball arises. On the other hand, the fiber which constitutes a web or more from 1.0 tangles, it results ill-behaved, and a pilling arises. It is the range of 0.6-0.8 from a viewpoint that intensity and front-face nature are excellent preferably [ both ].

[0018] The desirable manufacturing method of the polyester super-thin fiber web of this invention is explained below based on a drawing. After a suitable filter is therefore filtered, the polymer style by which melting was carried out within the extruder is led to the melting polymer induction (2) of a melt blow die (1), and is breathed out from an orifice-like nozzle (3) after that. The heating gas introduced into heating gas induction (4) simultaneously with it is led to the heating gas jet slit (5) formed of the melt blow die (1) and the lip (6), it blows off from here, this is \*\*-ized in the aforementioned \*\*\*\* polymer liquid, and super-thin fiber is formed. Subsequently, the uptake of this is carried out to the shape of a sheet, and a web is formed.

[0019] In this invention, it is important to make melting knockout temperature of polymer into 260-300 degrees C. If 300 degrees C is exceeded, polymer will cause acceleration of heat deterioration and hydrolysis etc., melt viscosity will fall, the intensity of the web obtained will fall, and also the fall of generation of a polymer ball, eyes spots, and dyeing robustness is caused. Next, it is important to make into 270-320 degrees C temperature of the elevated-temperature high-speed gas style spouted in order to blow away and make a polymer style super-thin in this invention. At less than 270 degrees C, since the cooling effect over a \*\*\*\* polymer style becomes excessive, \*\*-izing [ of a polymer style ] will become inadequate, a polymer ball will increase, and the quality of a web will be reduced sharply. On the other hand, if 320 degrees C is exceeded, the temperature of melting polymer induction (2) and an orifice-like nozzle (3) will exceed 300 degrees C with the heat transfer from this gas. Preferably, it is 270-290 degrees C. As blow gas to be used, degradation of polymer has steam and advantageous air also from a cost side few.

[0020] The injection pressure of the elevated-temperature high-speed gas spouted in this invention is 2kg [ 0.3-4 //cm ]. It is desirable to set it as the range. In addition, an injection pressure is the value measured at the point near the lip (6) of heating gas induction (4). This injection pressure is 0.3 kg/cm<sup>2</sup>. Since the polymer \*\*-ized energy of a jet gas becomes small in being the following, \*\*-izing becomes poor. Therefore, the fall of flexibility and eyes spots happen. On the other hand, it is 4 kg/cm<sup>2</sup>.

Above, a part of web by which coloring nature and robustness fell by \*\*-ization of polymer progressing although the intensity of a web became strong and it became a desirable direction, and the uptake was carried out since the force of a jet gas was too strong will be able to wind up, and surface grace will be inferior. Such a phenomenon becomes easy to happen in polytrimethylene terphthalate, although not generated in a polyethylene terephthalate.

[0021]

[The operation form of invention] Although an example etc. is given and this invention is hereafter explained more to a detail, needless to say, this invention is not limited at all by the example etc. In addition, the main measured value in an example was measured by the following methods.

(1) The limiting viscosity [eta] of \*\*\*\*\* is a value calculated based on the following definition formula.

$$[\eta] = \lim_{C \rightarrow 0} \frac{1}{C} (\eta_{sp} - 1)$$

$\eta_{sp}$  in a C->0 definition formula is the value which broke the 35-degree C viscosity of the diluted solution of the sample which dissolved by o-chlorophenol of 98% or more of purity by viscosity of the above-mentioned solvent itself measured at the same temperature, and is defined as relative viscosity. Moreover, C is a solute weight value by the gram unit in the 100ml of the above-mentioned solutions.

[0022] (2) Using DSC by the melting point SEIKO electronic company, by the programming rate of 20 degrees C/min, it is nitrogen air current Shimonaka of 100 ml/min, and measured. Here, peak value of the peak of dissolution was made into the melting point.

(3) Chloroform extracted the polyester resin constituent and polyester fiber into which PTT oligomer turned minutely [ fixed quantity ] for 50 hours using the Soxhlet extractor, and weight % to the sample using the oligomer contained in the obtained

residue showed them.

[0023] (4) It asked for a polyester resin constituent and the with a molecular weight of 300 or less contained in fiber organic substance from the chloroform liquid obtained in the structure determination and the fixed quantity (3) of the with a molecular weight of 300 or less organic substance. It analyzed using the gas chromatography equipped with the capillary column. The used column used two sorts, a silicon system and a polyethylene-glycol system. It asked for the concentration to the sample which the weighing capacity created the calibration curve to structure determination, and was used for it about each separated component using the mass spectrum (GC-MS) in ppm.

[0024] (5) About ten places with the arbitrary diameter sample of average fiber, ten-sheet photography was performed by one 2000 times the scale factor of this with the electron microscope. About one photograph, the diameter of ten arbitrary fiber was measured and ten photographs were followed in this. A total of 100 diameter measured value of fiber was calculated, and the average was calculated.

(6) The 10cmx10cm sample was continuously cut off over the width direction of an eyes spots web, and this weight was measured. The difference R of the average A of the value, maximum, and the minimum value was searched for, and eyes spots were measured by the following formula.

Eyes spots =R/Ax100. [0025] (7) It evaluated using the degree cantilever method of adaptability (45 degrees). It is shown that flexibility is so high that a numeric value is small.

(8) In the cheesecloth, the 400g load was hung and the wear \*\*\*\*\* sheet was worn out 500 times. The front face was observed and surface change was observed.

(9) Hyperchromicity and \*\*\*\*\* KAYARON polyester blue 3RSF (Nippon Kayaku Co., Ltd. make : tradename) used owf 10%, dace spar tangent line(Akinari chemistry company make : tradename) 1g/l., a bath ratio 1:50, and pH were made the dispersant 5, and dyeing was performed in the state of boil for 1 hour. Coloring nature evaluated the thickness of a color by the 1-5th class. In this case, it is shown that hyperchromicity is excellent, so that a number goes up. The dyeing object was rubbed 200 times, hanging a 200g load by the wet cheesecloth using a \*\*\*\* type friction tester, after drying a dyeing object, and the 1-5th class estimated the color projection to a cheesecloth. In this case, it is shown that there are so few color changes that a number goes up.

[0026] (Examples 1 and 2) The dimethyl terephthalate and 1 and 3-propanediol were taught by the mole ratio of 1:2, the mixture (9:1) of the calcium acetate equivalent to 0.1wt(s)% of the amount of theoretical polymer and a cobaltous acetate was added, the temperature up was carried out gradually, and the ester exchange reaction was completed at 240 degrees C. the obtained ester-interchange object -- titanium tetrapod butoxide -- 0.1wt(s)% of the amount of theoretical polymer -- it added and was made to react at 270 degrees C for 2 hours The limiting viscosity of the obtained polymer was 0.7. The amount of oligomer was 0.1wt(s)% , the with a molecular weight of 300 or less organic amount of resources was 330 ppm, and the melting point was 234 degrees C. Spinning did not have the thread breakage, either and did not have a fluff, either.

[0027] Dissolve the obtained polymer at 290 degrees C using an extruder, melting polymer is made to breathe out from the orifice of 0.3mmphi located in a line with the 1500-piece single tier in 1mm pitch, and it is continuously accumulated on the uptake side you made it located in 60cm under a die orifice and where it moves, and is eyes 200 g/m2. It rolled round as a random web so that it might become. The melt blow conditions of examples 1 and 2 and the acquired physical properties of a web were shown in Table 1.

[0028] Next, they are 30 kg/cm2 from the orifice of the 0.2mm diameter arranged in a straight line in 3mm pitch while putting this random web on the wire gauze and drawing in by degree of vacuum 50mmHg from the lower part. It spouts [ which is spouted continuously / high-speed ] out all over a sheet, it is put in a pressure, and, subsequently they are 10 kg/cm2. It processed similarly by the pressure. The physical properties of the obtained sheet were also shown in Table 1. the web and sheet which were obtained are excellent in flexibility and wear nature, and can be dyed a dark color by the ordinary pressure, and there are few polymer balls -- it excelled

[0029] (Examples 1 and 2 of comparison) It experimented by changing melt blow conditions into a Table 1 publication using the polymer manufactured in the example 1. The physical properties of the obtained web and a sheet were shown in Table 1. In any case, since the diameter of average fiber separated from the range of this invention, it was crude.

(Examples 3 and 4 of comparison) The same experiment as an example 1 was conducted using the polyethylene terephthalate (example 3 of comparison), and the polybutylene terephthalate (example 4 of comparison). It experimented by changing melt blow conditions into a Table 1 publication. The physical properties of the obtained web and a sheet were shown in Table 1. In the example 3 of comparison, coloring nature is bad and it turns out in the example 4 of comparison that \*\*\*\*\* is bad.

[0030]

[Table 1]

実施例	メルトブロー条件		ケープ物性				シート物性					
	スチーム温度	スチーム圧力	平均纖維径	極限粘度	6%+5%の発生	目付班	見かけ密度	引張強度	柔軟度	耐耗性	発色性	摩擦接着牢性
	℃	kg/cm <sup>2</sup>	μm		%	kg/cm <sup>3</sup>	kg/cm	mm		級	級	
1	288	1.8	4.0	0.68	ほとんどなし	4	0.25	2.1	4.5	良好	5	3-4
2	280	1.8	3.5	0.67	なし	5	0.24	2.0	4.4	“	5	3-4
比較例												
1	280	6.0	0.7	0.48	多い	5	0.30	2.3	4.0	不良	4	2
2	280	0.2	10.2	0.60	ほとんどなし	8	0.35	1.7	8.1	不良	6	3-4
3	300	8.0	3.5	0.58	なし	5	0.22	2.6	5.8	不良	3	4
4	280	1.8	3.2	0.68	少ない	8	0.25	1.8	4.2	普通	5	1-2

[0031] (Example 5 of comparison) The example 1 was repeated except having used titanium tetrapod butoxide 0.1wt% instead of the mixture of a calcium acetate and a cobaltous acetate as an ester-interchange catalyst. The amount of oligomer of the obtained polymer was 3.5wt(s)%, the with a molecular weight of 300 or less organic amount of resources was 1700 ppm, and the melting point was 233 degrees C. Although spinning was performed using this polymer, the white organic substance deposited in the lip side, and it was admitted in response to the influence that white powder was mixed in a sheet. When white powder was analyzed, it turns out that it is dimerized annular oligomer.

[0032]

[Effect of the Invention] According to this invention, by the melt blowing method, it can excel in flexibility and wear nature, and can be dyed a dark color by the ordinary pressure, and the polytrimethylene terephthalate super-thin fiber web by the melt blowing method with few polymer balls can be offered. This web is useful on objects for garments, such as an artificial leather base fabric, a filter, a tape, a disposable diaper, a PAPPU agent base fabric, work clothes, a wiping cross, a keeping-warm cotton pad, a padding cloth, a sheet, etc.

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[Translation done.]

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## Search Results - Record(s) 1 through 1 of 1 returned.

1. Document ID: JP 11107154 A

L1: Entry 1 of 1

File: DWPT

Apr 20, 1999

DERWENT-ACC-NO: 1999-330038

DERWENT-WEEK: 200009

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TITLE: Polyester fibre web for garments, filter, tape etc - consists of extra fine fibre made of poly:trimethylene terephthalate having predefined mean diameter and intrinsic viscosity

PRIORITY-DATA: 1997JP-0279377 (September 29, 1997)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
<u>JP 11107154 A</u>	April 20, 1999		006	D04H003/16

INT-CL (IPC): D01 F 6/62; D04 H 3/16

ABSTRACTED-PUB-NO: JP 11107154A

### BASIC-ABSTRACT:

NOVELTY - The web consists of extra fine fibers made of polytrimethylene terephthalate having mean diameter of 0.8-5  $\mu$ m and intrinsic viscosity of 0.4-1.0.

USE - For garments, artificial leather base fabric, filter, tape, disposable diaper, wiping cloth etc.

**ADVANTAGE** - The fiber web has excellent softness and wear property.

Full Title     **CLS.1**     **SEQ.1 ATT.1**    

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Term	Documents
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**WEST**[Generate Collection](#)[Print](#)**Search Results - Record(s) 1 through 7 of 7 returned.****□ 1. Document ID: US 6057256 A**

L1: Entry 1 of 7

File: USPT

May 2, 2000

US-PAT-NO: 6057256

DOCUMENT-IDENTIFIER: US 6057256 A

**\*\* See image for Certificate of Correction \*\***

TITLE: Web of biocomponent blown fibers

DATE-ISSUED: May 2, 2000

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Krueger; Dennis L.	St. Paul	MN		
Dyrud; James F.	St. Paul	MN		

US-CL-CURRENT: 442/400; 264/172.14, 428/373, 428/374, 442/351, 442/357, 442/361,  
442/362

## ABSTRACT:

Fibrous webs of bicomponent fibers are made by extruding a layered molten mass through a row of side-by-side orifices into a high-velocity gaseous stream. Bicomponent fibers of small size can be obtained, and the webs offer unique properties.

12 Claims, 5 Drawing figures

Exemplary Claim Number: 1

Number of Drawing Sheets: 1

[Full](#) | [Title](#) | [Abstract](#) | [Front](#) | [Review](#) | [Classification](#) | [Date](#) | [Reference](#) | [Sequences](#) | [Attachments](#) | [Claims](#) | [RIMC](#) | [Draw Desc](#) | [Image](#)**□ 2. Document ID: US 5364694 A**

L1: Entry 2 of 7

File: USPT

Nov 15, 1994

US-PAT-NO: 5364694

DOCUMENT-IDENTIFIER: US 5364694 A

**\*\* See image for Certificate of Correction \*\***

TITLE: Polyethylene terephthalate-based meltblown nonwoven fabric ad process for producing the same

DATE-ISSUED: November 15, 1994

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Okada; Hiromasa	Kurashiki			JP
Asano; Shoji	Kurashiki			JP

US-CL-CURRENT: 442/347; 156/166, 156/167, 264/172.18, 264/177.17, 264/DIG.26,  
428/373, 428/903, 442/363, 442/400

## ABSTRACT:

Provided is a polyethylene terephthalate-based meltblown nonwoven fabric comprising a mixed polymer comprising 75 to 98% by weight of polyethylene terephthalate and 2 to 25% by weight of a polyolefin. The meltblown fabric has excellent thermal resistance, dimensional stability, strength and hand. Also provided is a process for producing a polyethylene terephthalate-based meltblown nonwoven fabric, which comprises melt blowing a mixed polymer comprising 75 to 98% by weight of polyethylene terephthalate and 2 to 25% by weight of a polyolefin. It is preferable that the melt blowing is conducted at a single orifice throughput of 0.2 to 1.0 g/min and under an air-jet pressure of 0.1 to 1.0 kg/cm.sup.2.

13 Claims, 0 Drawing figures

Exemplary Claim Number: 1

[Full](#) | [Title](#) | [Citation](#) | [Front](#) | [Review](#) | [Classification](#) | [Date](#) | [Reference](#) | [Sequences](#) | [Attachments](#) | [NewC](#) | [Draw Desc](#) | [Image](#)

### 3. Document ID: US 4795668 A

L1: Entry 3 of 7

File: USPT

Jan 3, 1989

US-PAT-NO: 4795668

DOCUMENT-IDENTIFIER: US 4795668 A

**\*\* See image for Certificate of Correction \*\***

TITLE: Bicomponent fibers and webs made therefrom

DATE-ISSUED: January 3, 1989

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Krueger; Dennis L.	St. Paul	MN		
Meyer; Daniel E.	St. Paul	MN		

US-CL-CURRENT: 428/174; 428/221, 428/323, 428/332, 428/339, 428/357, 428/372,  
428/373, 428/401

## ABSTRACT:

Biocomponent fibers, and webs made therefrom, are taught in which one component of the fibers is a crystallizable material. When a web of the fibers is heated in a mold above the temperature at which crystallization occurs, the fibers tend to be set in the position they are held in the mold. A preferred fiber comprises blown fibers comprising as one component amorphous, crystallizable polyethylene terephthalate, and as the other component polypropylene. A web of such fibers not only becomes formed into a shape-retaining form by crystallization during the molding process, but also can be further established in its shape-retaining character by heating the web above the softening point of the polypropylene, whereupon the fibers become bonded at their points of intersection.

17 Claims, 5 Drawing figures  
Exemplary Claim Number: 1  
Number of Drawing Sheets: 1

[Full](#) | [Title](#) | [Citation](#) | [Front](#) | [Review](#) | [Classification](#) | [Date](#) | [Reference](#) | [Sequences](#) | [Attachments](#) | [RMC](#) | [Draw Desc](#) | [Image](#)

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#### □ 4. Document ID: US 4729371 A

L1: Entry 4 of 7

File: USPT

Mar 8, 1988

US-PAT-NO: 4729371

DOCUMENT-IDENTIFIER: US 4729371 A

TITLE: Respirator comprised of blown bicomponent fibers

DATE-ISSUED: March 8, 1988

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Krueger; Dennis L.	Hudson	WI		
Dyrud; James F.	New Richmond	WI		

US-CL-CURRENT: 128/206.19; 428/359, 428/362, 428/372, 428/374, 442/352, 442/357

ABSTRACT:

Fibrous webs of bicomponent fibers are made by extruding a layered molten mass through a row of side-by-side orifices into a high-velocity gaseous stream. Bicomponent fibers of small size can be obtained, and the webs offer unique properties.

8 Claims, 5 Drawing figures

Exemplary Claim Number: 1

Number of Drawing Sheets: 1

[Full](#) | [Title](#) | [Citation](#) | [Front](#) | [Review](#) | [Classification](#) | [Date](#) | [Reference](#) | [Sequences](#) | [Attachments](#) | [RMC](#) | [Draw Desc](#) | [Image](#)

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#### □ 5. Document ID: US 4657804 A

L1: Entry 5 of 7

File: USPT

Apr 14, 1987

US-PAT-NO: 4657804

DOCUMENT-IDENTIFIER: US 4657804 A

**\*\* See image for Certificate of Correction \*\***

TITLE: Fusible fiber/microfine fiber laminate

DATE-ISSUED: April 14, 1987

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Mays; Alfred T.	East Windsor	NJ		
Yang; Ching-Yun M.	East Windsor	NJ		

US-CL-CURRENT: 428/212; 156/182, 156/244.24, 156/244.27, 156/278, 156/280,  
156/308.2, 206/438, 206/439, 206/524.2, 206/524.6, 206/811, 220/DIG.11, 229/5.81,  
229/5.84, 229/68.1, 229/75, 428/315.9, 428/317.3, 428/317.5, 428/317.7, 428/76,  
442/346, 442/361, 442/381

ABSTRACT:

A water-impervious, smooth-surfaced, gas-permeable, bacterial barrier, repellent treated, laminated material is described. A preferred embodiment comprises a ply of hydrophobic microfine fibers fuse bonded to a layer of conjugate fibers having a low melting sheath and a high melting core. The ply of hydrophobic microfine fibers is low melting. The sheaths of the conjugate fibers have been fuse bonded to the hydrophobic microfine fibers at a temperature below the melt temperature of the cores of the conjugate fibers so that the cores retain their initial fiber-like integrity. The laminated material is preferably impregnated with both a repellent binder and a repellent finish to secure good repellency, lamination and peelability.

29 Claims, 0 Drawing figures

Exemplary Claim Number: 1

[Full](#) | [Title](#) | [Citation](#) | [Front](#) | [Review](#) | [Classification](#) | [Date](#) | [Reference](#) | [Sequences](#) | [Attachments](#) | [KMD](#) | [Drawn Desc](#) | [Image](#)

■ 6. Document ID: US 4547420 A

L1: Entry 6 of 7

File: USPT

Oct 15, 1985

US-PAT-NO: 4547420

DOCUMENT-IDENTIFIER: US 4547420 A

TITLE: Bicomponent fibers and webs made therefrom

DATE-ISSUED: October 15, 1985

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Krueger; Dennis L.	Hudson	WI		
Meyer; Daniel E.	Stillwater	MN		

US-CL-CURRENT: 442/347; 264/172.14, 264/172.17, 264/172.18, 428/373, 442/362

ABSTRACT:

Bicomponent fibers, and webs made therefrom, are taught in which one component of the fibers is a crystallizable material. When a web of the fibers is heated in a mold above the temperature at which crystallization occurs, the fibers tend to be set in the position they are held in the mold. A preferred fiber comprises blown fibers comprising as one component amorphous, crystallizable polyethylene terephthalate, and as the other component polypropylene. A web of such fibers not only becomes formed into a shape-retaining form by crystallization during the molding process, but also can be further established in its shape-retaining character by heating the web above the softening point of the polypropylene, whereupon the fibers become bonded at their points of intersection.

11 Claims, 5 Drawing figures

Exemplary Claim Number: 1

Number of Drawing Sheets: 1

[ Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments ] [ KAMC | Draw Desc | Image ]

## □ 7. Document ID: US 4508113 A

L1: Entry 7 of 7

File: USPT

Apr 2, 1985

US-PAT-NO: 4508113

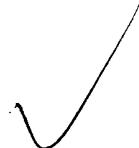
DOCUMENT-IDENTIFIER: US 4508113 A

TITLE: Microfine fiber laminate

DATE-ISSUED: April 2, 1985

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Malaney, Frank E.	Warren	NJ		



US-CL-CURRENT: 128/849; 128/206.19, 428/212, 442/340, 442/361, 442/389

ABSTRACT:

A water impervious laminated material is described. A preferred embodiment comprises a three-ply hydrophobic microfine fiber structure sandwiched between and fuse bonded to two layers of conjugate fibers having a low melting sheath and a high melting core. The inner ply of the hydrophobic microfine fiber structure is relatively high melting while the two outer plies of the hydrophobic microfine fiber structure film are low melting. The sheaths of the conjugate fibers have been fuse bonded to the hydrophobic microfine fiber structure at a temperature below the melt temperature of the cores of the conjugate fibers so that the cores retain their initial fiber-like integrity.

14 Claims, 2 Drawing figures

Exemplary Claim Number: 1

Number of Drawing Sheets: 1

[ Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments ] [ KAMC | Draw Desc | Image ]

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**WEST**[Generate Collection](#)[Print](#)**Search Results - Record(s) 1 through 1 of 1 returned.****1. Document ID: JP 02104755 A**

L2: Entry 1 of 1

File: DWPI

Apr 17, 1990

DERWENT-ACC-NO: 1990-161475

DERWENT-WEEK: 199021

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TITLE: Fine polyester fibre web - comprises polyethylene terephthalate and is obtd. by melt-blown polyester

PRIORITY-DATA: 1989JP-0192716 (January 1, 1989), 1979JP-0046037 (April 17, 1979)

## PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
<u>JP 02104755 A</u>	April 17, 1990		000	

INT-CL (IPC): D04H 1/72

ABSTRACTED-PUB-NO: JP 02104755A

## BASIC-ABSTRACT:

A web consisting of a very fine polyester fibre is obtd. by melt-blown of a polyester polymer and the very fine fibre has ave. fibre 0.8-5.0 mm and has intrinsic viscosity 0.45-0.80. The very fine web is composed of polyethylene terephthalate. A web consisting of a very fine polyester fibre in which Y value of the fibre web is at 2.5-7.0. The polyester polymer to compose the polyester fibre has intrinsic viscosity 0.50-0.90 and the intrinsic viscosity of the fine fibre (A) and a polyester polymer (B) satisfy the equation: A is greater than or equal to B - 0.2. When the very fine fibre is measured by differential scanning colorimetry(DSC), crystallisation peak is small and melting peak is multiple. Unevenness of web wt. in the width direction is less than 10%. The web is not polymer nap.

USE/ADVANTAGE - The web has good strength and dyeing properties and is pref. used as a ground cloth for a raised man-made leather.

[Full](#) | [Title](#) | [Citation](#) | [Front](#) | [Review](#) | [Classification](#) | [Date](#) | [References](#) | [Sequences](#) | [Attachments](#) | [Claims](#) | [R&D](#) | [Draw Desc](#) | [Image](#)[Generate Collection](#)[Print](#)

**WEST****Generate Collection****Print****Search Results - Record(s) 1 through 1 of 1 returned.** **1. Document ID: US 4518744 A**

L1: Entry 1 of 1

File: USPT

May 21, 1985

US-PAT-NO: 4518744

DOCUMENT-IDENTIFIER: US 4518744 A

TITLE: Process of melt spinning of a blend of a fibre-forming polymer and an immiscible polymer and melt spun fibres produced by such process

DATE-ISSUED: May 21, 1985

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Brody; Harry	Harrogate			GB2

US-CL-CURRENT: 525/184; 264/176.1, 264/211, 264/349, 264/DIG.29

## ABSTRACT:

A process of melt spinning a fibre-forming thermoplastic polymer, more particularly polyethylene terephthalate, polyhexamethylene adipamide or polypropylene, at a minimum wind up speed of 2 kilometers per minute in which there is added to the fibre-forming polymer, between 0.1% and 10% by weight of another polymer which is immiscible in a melt of the fibre-forming polymer, such other polymer having a particle size of between 0.5 and 3 microns in the melt with the fibre-forming polymer immediately prior to spinning and novel melt spun fibres produced by such a process and in which the other polymer is in the form of microfibrils.

5 Claims, 9 Drawing figures

Exemplary Claim Number: 1

Number of Drawing Sheets: 8

Full

Title

CLS.1

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ATT.1

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4518744S	0
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**WEST**[Generate Collection](#)[Print](#)**Search Results - Record(s) 1 through 3 of 3 returned.** **1. Document ID: US 5225237 A**

L1: Entry 1 of 3

File: USPT

Jul 6, 1993

US-PAT-NO: 5225237

DOCUMENT-IDENTIFIER: US 5225237 A

TITLE: Building sheets of cement material reinforced with plastics mesh and glass fibers

DATE-ISSUED: July 6, 1993

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Magnani; Silvio	Canneto Pavese			IT

US-CL-CURRENT: 442/57; 106/754, 428/703, 52/782.1

## ABSTRACT:

Building sheets consisting of cement, inert materials and additives, and reinforced with plastics mesh and alkali-resistant glass fibers of short and/or continuous type, including a number of superposed elementary layers consisting of a mixture of cement, inert materials and additives and each comprising as reinforcement material a plastics mesh or glass fibers. The apparatus for preparing the building sheets includes a frame, a conveyor belt, support rollers and a slide surface for the conveyor belt, an inversion roller and a drive roller, a possible feeder for a continuous support web, a series of plastics mesh feeders, a series of feeders for glass fiber originating from bobbins, a series of cement mix metering pumps, a series of cement mix distributors and a series of smoothing devices.

13 Claims, 4 Drawing figures

Exemplary Claim Number: 1

Number of Drawing Sheets: 3

[Full](#) | [Title](#) | [Citation](#) | [Front](#) | [Review](#) | [Classification](#) | [Date](#) | [Reference](#) | [Sequences](#) | [Attachments](#) | [Claims](#) | [RQMC](#) | [Draw Desc](#) | [Image](#) **2. Document ID: US 4378405 A**

L1: Entry 2 of 3

File: USPT

Mar 29, 1983

US-PAT-NO: 4378405

DOCUMENT-IDENTIFIER: US 4378405 A

TITLE: Production of building board

DATE-ISSUED: March 29, 1983

## INVENTOR-INFORMATION:

NAME Pilgrim; Thomas A.	CITY Edwalton	STATE	ZIP CODE	COUNTRY GB2
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US-CL-CURRENT: 428/322.7; 156/42, 428/703

## ABSTRACT:

A building board comprises a core 31 of set cementitious material, such as gypsum, faced on at least one side by a fabric or web 33 of mineral fibres embedded in the face of the core and a continuous film 32 of the cementitious material having a higher density and lower porosity than the core extending over the outer face of the fabric. The film may have a smooth surface or a desired textured or figured surface. The preferred fabric or web 33 is a non-woven glass fibre tissue and it is preferred that a continuous film 32 of gypsum at the surface should not exceed 2 mm in thickness. The board is made by bringing the fabric or web into contact with the respective face of a layer of an aqueous slurry of gypsum plaster or other cementitious material, and vibrating the layer of slurry in contact with the fabric or web until slurry penetrates the web and the latter is completely embedded. The process can be carried out continuously by supporting the slurry between flexible belts which are vibrated by mechanical action applied to their faces remote from the slurry. Boards accordingly to the invention can have greater strength and/or greater resistance to fire than conventional paper-faced gypsum board.

16 Claims, 2 Drawing figures

Exemplary Claim Number: 10

Number of Drawing Sheets: 1

<input type="checkbox"/> Full	<input type="checkbox"/> Title	<input type="checkbox"/> Citation	<input type="checkbox"/> Front	<input type="checkbox"/> Review	<input type="checkbox"/> Classification	<input type="checkbox"/> Date	<input type="checkbox"/> Reference	<input type="checkbox"/> Sequences	<input type="checkbox"/> Attachments	<input type="checkbox"/> Claims	<input type="checkbox"/> KWD	<input type="checkbox"/> Draw Desc	<input type="checkbox"/> Image
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 3. Document ID: US 3944698 A

L1: Entry 3 of 3

File: USPT

Mar 16, 1976

US-PAT-NO: 3944698

DOCUMENT-IDENTIFIER: US 3944698 A

TITLE: Gypsum wallboard and process for making same

DATE-ISSUED: March 16, 1976

## INVENTOR-INFORMATION:

NAME Dierks; Robert P.	CITY Des Plaines	STATE	ZIP CODE	COUNTRY
Rillie; Robert J.	Des Plaines	IL		

US-CL-CURRENT: 428/219; 156/42, 428/294.7, 428/312.4, 428/535, 428/703

## ABSTRACT:

A specially prepared fiber reinforcement and improved gypsum wallboard are disclosed. The fiber reinforcement includes a multiplicity of relatively long fibers which are disposed at the interface of the core and cover sheets of the wallboard and are adhesively bonded to the cover sheets and incorporated predominantly into the portion of the core immediately adjacent to the cover sheets.

14 Claims, 4 Drawing figures  
Exemplary Claim Number: 1  
Number of Drawing Sheets: 2

[ Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Claims | IOMC | Drawn Desc | Image ]

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